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EFFECT OF HUMIDITY AND A WETTABILITY ADDITIVE ON POLYPHENYL ETHER BOUNDARY LUBRICATION OF STEEL IN AIR AND NITROGEN TO 350° C

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16. Abstract

A pin-on-disk sliding friction apparatus was used to determine the effect of humidity and a wettability additive on boundary lubrication of steel in air and nitrogen with a polyphenyl ether. Poor wettability was observed for the ether in dry nitrogen from 150° to 200° C (302° to 392° F) and high wear also occurred in this temperature range. The wettability additive improved the wetting and decreased wear of the ether in dry nitrogen. It increased wear in wet air and had little effect on wear in wet nitrogen and dry air. A relative humidity of 50 percent decreased wear in nitrogen and had little effect on wear in air.

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EFFECT OF HUMIDITY AND A WETTABILITY ADDITIVE ON POLYPHENYL ETHER BOUNDARY LUBRICATION OF STEEL IN AIR AND NITROGEN TO 350° C

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SUMMARY

A pin-on-disk sliding friction apparatus was used to determine the effect of humidity and a wettability additive on boundary lubrication of steel in air and nitrogen with a five-ring polyphenyl ether. The test atmospheres were (1) wet air (RH 50%), (2) wet nitrogen (RH 50%), (3) dry air (<100 ppm H_2O), and (4) dry nitrogen (<20 ppm H_2O). Other conditions included a 1-kilogram load, 17-meter-per-minute surface velocity (100-rpm disk speed), 150° to 350° C (302° to 662° F) disk temperature range, and a 1-hour test duration. The wettability additive was of the halogenated organic acid type.

Poor wettability was observed for the polyphenyl ether in dry nitrogen from 150° to 200° C (302° to 392° F) and high wear also occurred in this temperature range.

The wettability additive improved the wetting characteristics of the polyphenyl ether in dry nitrogen. The additive decreased wear with the polyphenyl ether in dry nitrogen, increased wear in wet air, and had no or little effect on wear in wet nitrogen and dry air.

A relative humidity of 50 percent decreased wear in nitrogen and had little effect on wear in air.

INTRODUCTION

Polyphenyl ethers have been considered as possible high temperature lubricants for some time (refs. 1 to 3). This fluid type is attractive because of its oxidation stability, 340° C (644° F), thermal stability, 440° C (824° F), and radiation resistance (ref. 4). However, polyphenyl ethers have not performed well in some boundary lubrication and bearing fatigue studies (refs. 5 to 9).

Appeldoorn and Tao (ref. 10) have found that boundary lubrication with aromatic

compounds is greatly influenced by atmospheric oxygen and moisture. Reference 7 showed that rider wear for a polyphenyl ether in nitrogen increased an order of magnitude after fluid degassing.

Four ball tests (ref. 9) in nitrogen yielded maximum wear at 200° C (392° F). In vane pump studies (ref. 5) in dry nitrogen, polyphenyl ethers exhibited high wear at 219° C (425° F). Following the vane pump experiments it was observed that ether lubricants exhibited poor wettability on the test specimens. In this report, the terms poor wettability or poor wetting are used to denote a fluid having a finite contact angle on the solid surface.

A poor wetting condition could cause fluid starvation in the contact zone and prevent effective lubrication. Additives that can improve the wettability of a lubricant (i.e., decrease its contact angle) may improve its boundary lubrication characteristics. It is also possible that in a high speed bearing, where the cooling effect of the lubricant becomes important, an improvement in wettability may be beneficial by increasing local heat transfer rates. Because of the low speed conditions of this study, lubricant cooling effects should not be a factor.

The objective of this investigation was to determine the effect of humidity and a wettability additive on boundary lubrication of steel in air and nitrogen with a polyphenyl ether.

Experimental conditions with the pin-on-disk apparatus included a 1-kilogram load (initial hertz stress 1×10^{9} N/m^{2}), 17-meter-per-minute (m/min) surface velocity (100-rpm disk speed), 150° to 350° C (302° to 662° F) disk temperature range, a 1-hour test duration, and test atmospheres of wet and dry air and wet and dry nitrogen. Test specimens were made of consumable electrode vacuum melted (CVM) M-50 steel.

APPARATUS

The friction and wear test apparatus is shown in figure 1. The test specimens were contained inside a stainless steel chamber. The atmosphere was controlled with respect to oxygen and moisture content.

A 6.3-centimeter-diameter disk was placed in sliding contact with a 0.476-centimeter-radius hemispherically tipped rider. A surface velocity of 17 m/min (100 rpm) was maintained. A normal load of 1 kilogram was applied with a deadweight. Riders and disks were made of CVM M-50 tool steel, disk hardness was Rockwell C 62 to 64 and rider hardness was 56 to 58.

The disk was partially submerged in a pyrex cup containing the test lubricant. The disks were heated by induction. Bulk lubricant temperature was recorded with a thermo-

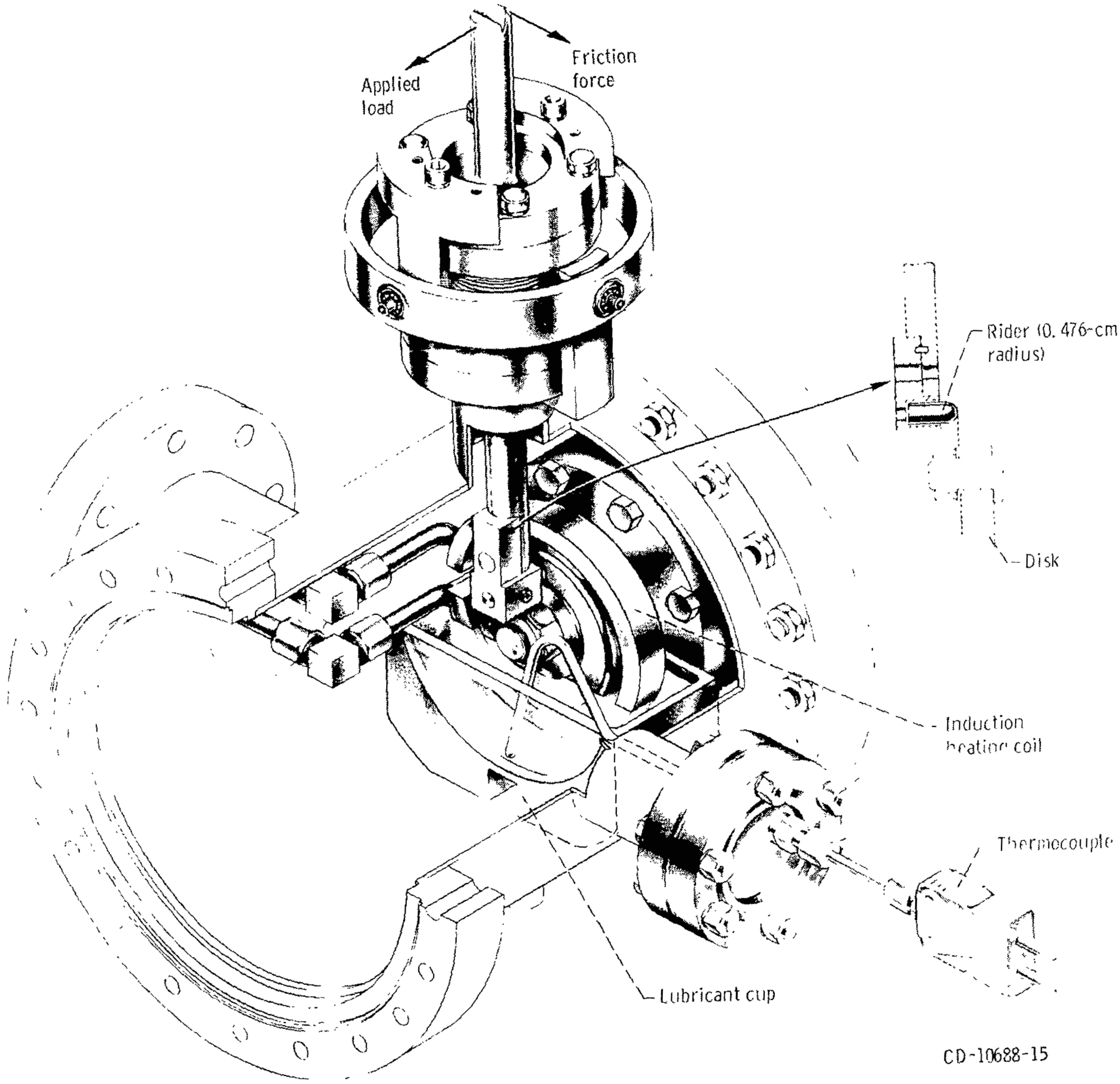


Figure 1. - Friction and wear apparatus.

couple. Disk temperature was monitored with an infrared pyrometer. Frictional force was measured with a strain gage and was recorded on a strip chart recorder.

ATMOSPHERE MONITORING AND CONTROL

The four atmospheres used in this study were (1) wet air, (2) wet nitrogen both at a relative humidity of 50±5 percent at 25° C (77° F), (3) dry air (<100 ppm H_2O), and (4) dry nitrogen (<20 ppm H_2O).

The relative humidity was monitored by a direct reading hygrometer accurate to ±1.5 percent. The low water concentrations were monitored by a moisture analyzer with an accuracy of ±10 parts per million.

Dry nitrogen was obtained directly from compressed gas cylinders. Dry air was obtained by drying and filtering service air. Wet air and nitrogen were obtained by bubbling the dry gases through a water reservoir. The relative humidity was controlled manually to 50±5 percent.

PROCEDURE

Disks and riders were made of CVM M-50 tool steel. They were ground and lapped to a surface finish of 4 to 8 microinches rms (10×10^{-8} to 20×10^{-8} m). Specimens were scrubbed with a paste of levigated alumina and water, rinsed with tap water and distilled water, then placed in a desiccator.

Lubricants were degassed at 150° C (302° F) under a vacuum. Dissolved oxygen concentration was measured with a polarographic probe and found to be less than 5 parts per million. No attempt was made to remove dissolved water.

The specimens were assembled and 70 milliliters (7×10^{-5} m^3) of lubricant were placed in the lubricant cup. The test chamber (3.7 liter volume or 3.7×10^{-3} m^3) was purged with the test atmosphere for 10 minutes at a flow rate in excess of 50 liters per hour (5×10^{-2} m^3/hr). The disk was heated by induction to test temperature while rotating and the rider loaded against the disk. Test atmosphere flow rate was reduced to 35 liters per hour (3.5×10^{-2} m^3/hr) and a 1-psig (6.9×10^{3}-N/m^2) pressure was maintained in the chamber. The lubricant was heated only by heat transfer from the disk and benefited by the cooling effect of the water circulating through the induction heating coil. Therefore, the bulk lubricant temperature (measured with a thermocouple) stabilized 100° to 150° C (180° to 270° F) below disk temperature.

Frictional force and bulk lubricant temperature were continuously recorded. Disk

temperature was continuously monitored. Tests were terminated at 1 hour, and rider wear scar diameter was recorded.

Disk Temperature Calibration

Disk temperatures were monitored with an infrared pyrometer. Instrument accuracy (at constant emissivity) was $\pm 1^{\circ}$ C (1.8° F) with a reproducibility of 0.25 percent of the temperature.

Disk emissivity was about 0.15 initially and increased to 0.2 to 0.3 during a test. This change introduced a large error (>17 percent) in temperature measurement. Therefore, a 2.5-centimeter-diameter spacer with a black oxide coating ($e \simeq 0.55$) was placed between the nut and disk. The pyrometer was then used to monitor the spacer temperature which was a function of disk temperature. Both temperatures were measured with thermocouples under static conditions and a calibration curve was obtained. A variation in spacer emissivity from 0.5 to 0.6 resulted in an error of less than 2 percent. Dynamic tests with and without test lubricant using a disk with a black oxide coating yielded results within 2.5 percent of the static calibration. Disk temperature was manually controlled to $\pm 5^{\circ}$ C (9° F).

Dip Cell Experiments

An elementary test was used to determine the wettability of the polyphenyl ether on M-50 tool steel. The procedure was similar to that employed by Bigelow, Pickett, and Zisman (ref. 11).

A flask containing 100 milliliters (1×10^{-4} m^3) of the lubricant was heated slowly (5° C/min). An M-50 tool steel specimen (4.1 by 1.3 by 0.24 cm) which was cleaned in the same manner as the disk and rider specimens was placed in the fluid. Starting at 150° C (302° F) the specimen was raised out of the fluid at 5° C intervals to a maximum of 250° C (482° F). The manner in which the fluid drained off the specimen was observed. The atmosphere above the fluid was controlled with respect to oxygen and moisture.

RESULTS AND DISCUSSION

Some properties of the five-ring polyphenyl ether used in this study appear in table I. The wettability additive was of the halogenated organic acid type and was present in a

TABLE I. - SOME PROPERTIES OF A FIVE-RING

POLYPHENYL ETHER

Property	
Kinematic viscosity, cS (m^2/sec)	
At 38° C (100° F)	360 (3.6×10^{-4})
At 99° C (210° F)	13 (1.3×10^{-5})
At 350° C (662° F)	0.72 (7.2×10^{-7})
Pour point, °C (°F)	5 (40)
Flash point, °C (°F)	288 (550)
Fire point, °C (°F)	350 (662)
Density at 38° C (100° F), g/ml (kg/m^3)	1.19 (1.19×10^3)
Thermal decomposition (isoteniscope), °C (°F)	443 (830)
Vapor pressure at 343° C (650° F), torr	12
Surface tension at 25° C (77° F), dynes/cm (N/cm)	50 (5×10^{-4})

concentration of 0.05 percent by weight. Disk temperature range was 150° to 350° C (302° to 662° F). The lower temperature was arbitrarily chosen and the upper limit was dictated by lubricant volatility.

In order to facilitate discussion of rider wear, the wear data have been divided into three arbitrary levels. These levels are (1) low wear which corresponds to a wear rate of less than 10^{-12} cubic meter per hour (wear scar diameter (WSD), 0.5 mm), (2) intermediate wear (wear rate between 10^{-12} and 10^{-11} m^3/hr), and (3) high wear (wear rate greater than 10^{-11} m^3/hr; WSD, 1.0 mm).

Effect of Atmosphere on Wear and Friction of Unlubricated Steel

Rider wear and friction coefficient for the unlubricated situation in all four atmospheres appear in figure 2. High wear (10^{-11} to 10^{-10} m^3/hr) occurred in wet and dry nitrogen. Very high wear (10^{-10} to 10^{-9} m^3/hr) occurred in wet and dry air. The friction coefficient in all four atmospheres varied from 0.6 to 0.8 at 150° C (302° F) and from 0.4 to 0.6 at 350° C (662° F).

Effect of Atmosphere on Base Fluid Wear

Dry air compared to dry nitrogen. - Rider wear for the base fluid in dry nitrogen and dry air appears in figures 3(a) and (b), respectively. Higher wear occurred in dry nitrogen from 150° to 250° C (302° to 482° F) and lower wear from 250° to 350° C (482° to 662° F) compared to the dry air situation.

The polyphenyl ether is a poor lubricant in dry nitrogen from 150° to 200° C (302° to 392° F). This agrees with the high wear obtained with this fluid in dry nitrogen in

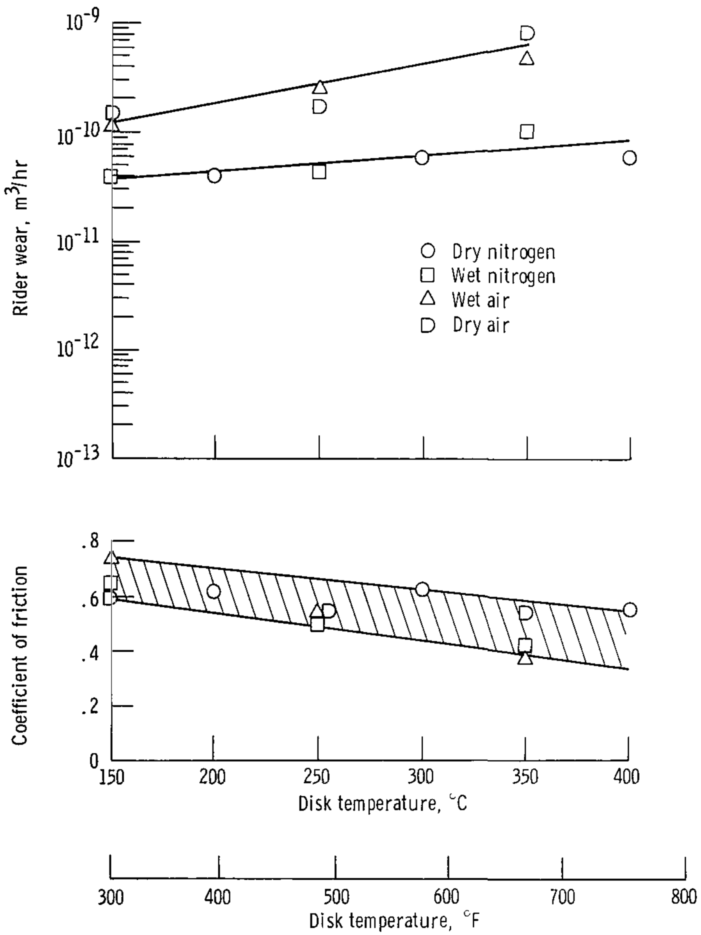
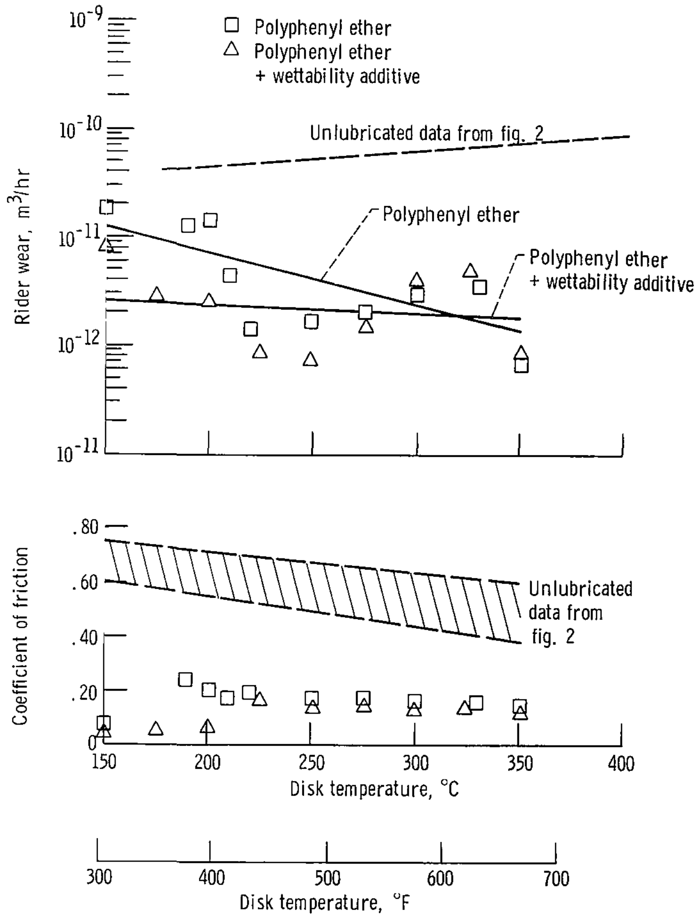
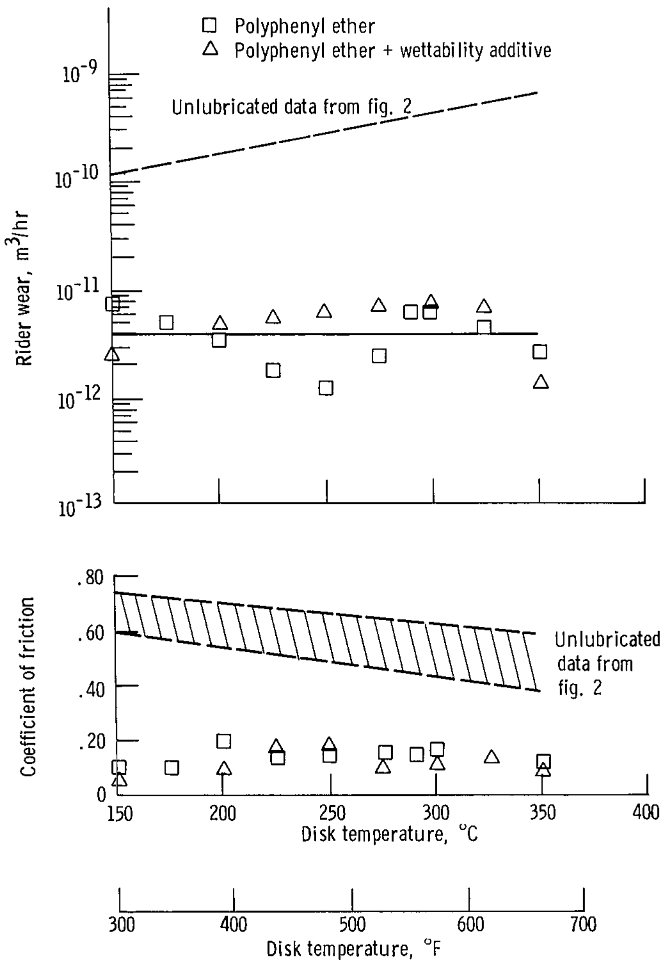


Figure 2. - Coefficient of friction and rider wear as a function of disk temperature for unlubricated M-50 steel in dry and wet nitrogen and in dry and wet air. Conditions: 1-kilogram load, 100-rpm disk speed, 17-meter-per-minute surface velocity, and 1-hour test duration.



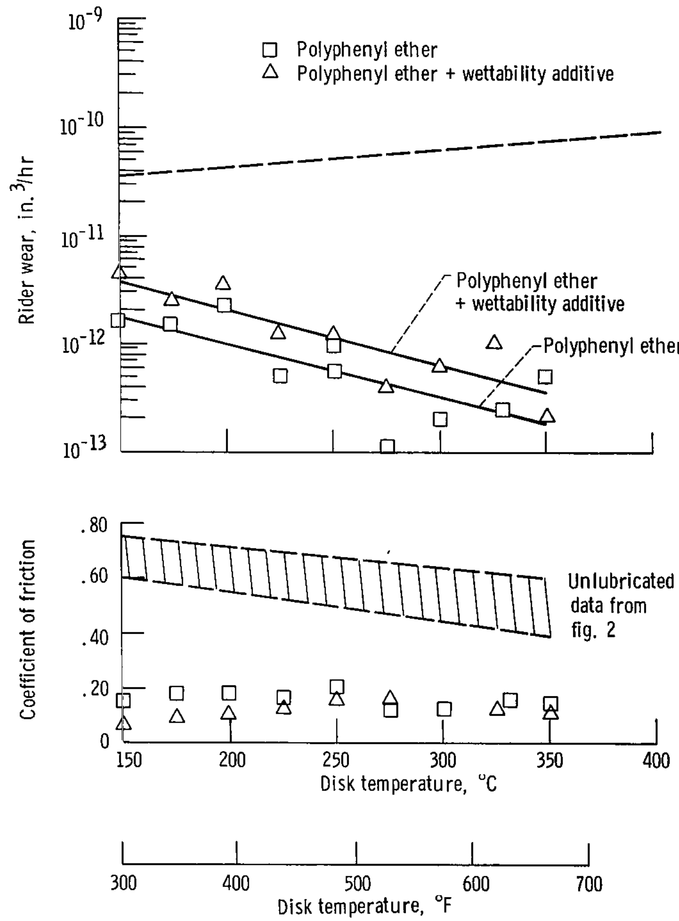
(a) Dry nitrogen.

Figure 3. - Coefficient of friction and rider wear as a function of disk temperature for polyphenyl ether with and without wettability additive. Conditions: 1-kilogram load, 100-rpm disk speed, 17-meter-per-minute surface velocity, and 1-hour test duration.



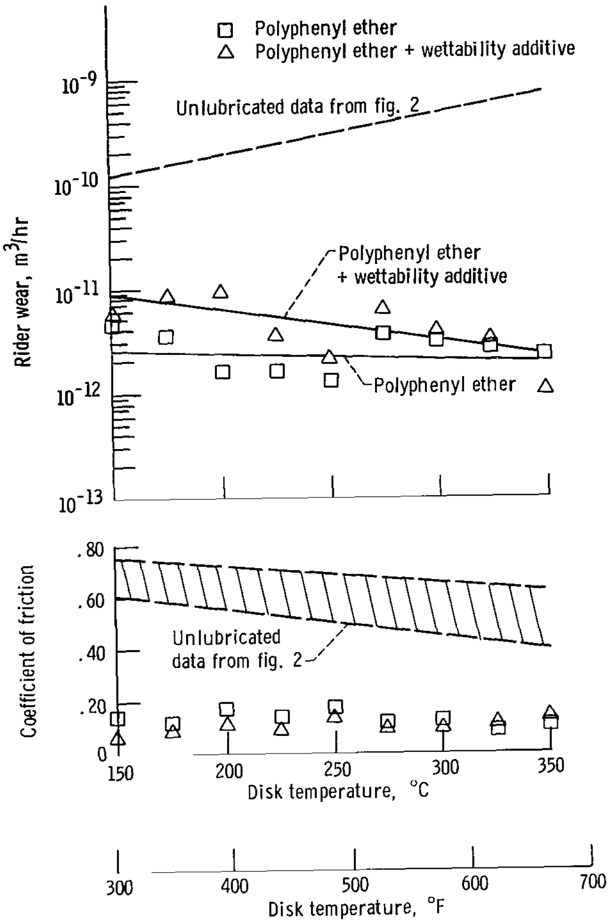
(b) Dry air.

Figure 3. - Continued.



(c) Wet nitrogen.

Figure 3. - Continued.



(d) Wet air.

Figure 3. - Concluded.

vane pump studies (ref. 5) and in four-ball tests (ref. 9). This also agrees with the suggestion of Appeldoorn and Tao indicating that aromatics are poor boundary lubricants in dry nonoxidizing conditions (ref. 10).

Effect of moisture. - Rider wear for the base fluid in wet nitrogen and wet air appears in figures 3(c) and (d), respectively. Substantially lower wear occurred in wet nitrogen compared to dry nitrogen. Slightly lower wear occurred in wet air compared to dry air. These results agree with the lower temperature results of references 10 and 12. Moisture apparently reduces wear of sliding parts lubricated with aromatic fluids. Figure 4 summarizes wear data in the four atmospheres at temperatures of 150°, 250°, and 350° C (302°, 482°, and 662° F).

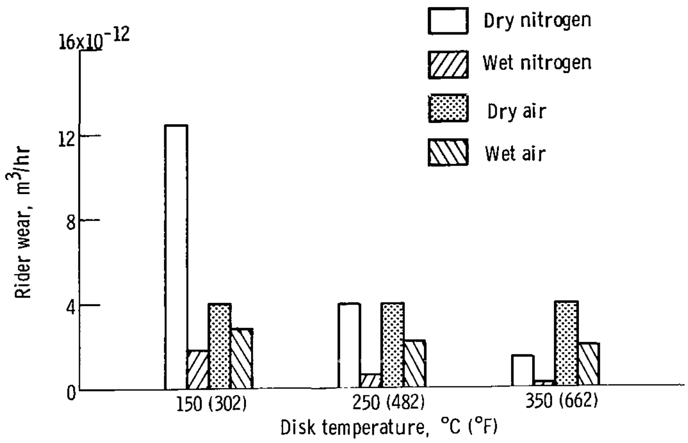


Figure 4. - Rider wear for polyphenyl ether in wet and dry nitrogen and in wet and dry air at disk temperatures of 150°, 250°, and 350° C (302°, 482°, and 662° F).

Effect of Wettability Additive on Rider Wear

Dry nitrogen. - Rider wear for the polyphenyl ether with additive in dry nitrogen appears in figure 3(a). The wettability additive reduced wear over most of the temperature range. The greatest wear reductions occurred in the 150° to 200° C (302° to 392° F) range.

Dry air. - Rider wear for the fluid with additive in dry air appears in figure 3(b). Essentially no difference in wear occurred for the fluid with and without additive in dry air.

Wet nitrogen. - Rider wear for the fluid with additive in wet nitrogen appears in figure 3(c). A small increase in wear occurred with the additive fluid in wet nitrogen. Figure 5 summarizes wear data for the fluid with and without additive in wet and dry

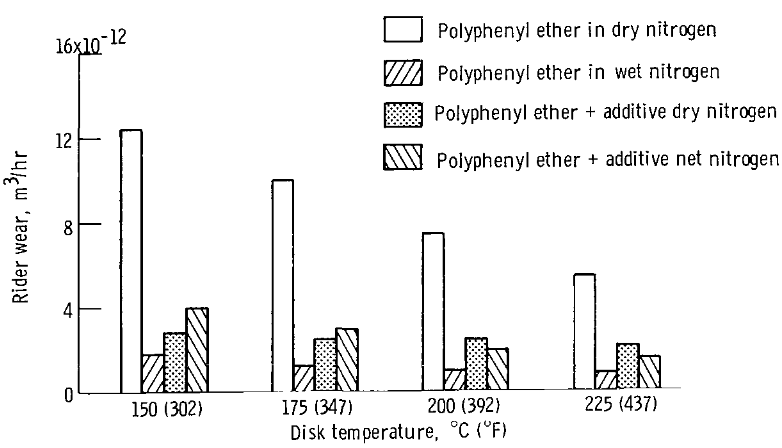


Figure 5. - Rider wear for polyphenyl ether with and without wettability additive in wet and dry nitrogen at four disk temperatures.

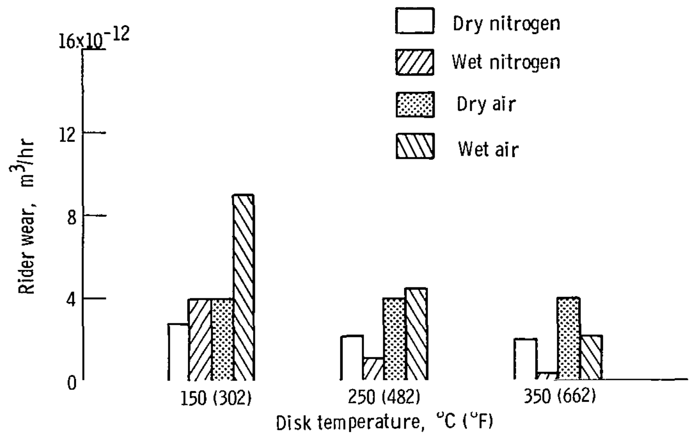


Figure 6. - Rider wear for polyphenyl ether with wettability additive in wet and dry nitrogen and wet and dry air at disk temperatures of 150°, 250°, and 350° C (302°, 482°, and 662° F).

nitrogen at disk temperatures of 150°, 175°, 200°, and 225° C (302°, 347°, 392°, and 437° F).

Wet air. - Rider wear for the fluid with additive in wet air appears in figure 3(d). Small increases in wear occurred over most of the temperature range. Figure 6 summarizes wear data for the fluid with additive in four atmospheres at temperatures of 150°, 250°, and 350° C (302°, 482°, and 662° F).

In general, the wettability additive was most effective in reducing wear in dry nitrogen from 150° to 200° C (302° to 392° F). It had little or no effect on wear in dry air and wet nitrogen and appeared to be detrimental in wet air. Table II summarizes wear data for all conditions.

"EP" or Antiwear Activity of Wettability Additive

Because of the chemical nature of the wettability additive, it may function as an antiwear or "EP" additive. There is no way to separate EP activity and wetting changes on wear data at this time. Future work with a more nonreactive wettability additive would help clarify the situation.

Dip Cell Experiments

Polyphenyl ethers exhibited poor wetting on test specimens in dry nitrogen in previous work (ref. 5). In these earlier studies, a four- and a five-ring polyphenyl ether

TABLE II. - SUMMARY OF WEAR RESULTS

High wear	Intermediate wear	Low wear
$>10^{-11}$ m^3/hr	Between 10^{-12} and 10^{-11} m^3/hr	$<10^{-12}$ m^3/hr
In dry nitrogen from 150° to 175° C (302° to 347° F) without additive	In wet and dry air 150° to 350° C (302° to 662° F) with and without additive	In wet nitrogen 260° to 350° C (500° to 662° F) with additive
	In dry nitrogen 150° to 350° C (302° to 662° F) with additive	In wet nitrogen 200° to 350° C (392° to 662° F) without additive
	In dry nitrogen 175° to 350° C (347° to 662° F) without additive	
	In wet nitrogen 150° to 200° C (302° to 392° F) without additive	
	In wet nitrogen 150° to 260° C (302° to 500° F) with additive	

exhibited very high wear in a vane pump at 219° C (425° F). In four-ball tests (ref. 9), 5P-4E yielded the highest wear at 200° C (392° F). In this investigation, the five-ring polyphenyl ether exhibited its highest wear in dry nitrogen from 150° to 200° C (302° to 392° F). Therefore, it was decided to study the wettability of 5P-4E from 150° to 250° C (302° to 482° F) in dry nitrogen with the dip cell apparatus.

A poor wetting condition was observed for the base fluid in dry nitrogen from 150° C (302° F) to approximately 195° C (383° F). Partial wetting occurred from 195° to 200° C (383° to 392° F). From 200° to 250° C (392° to 482° F) complete wetting occurred. The polyphenyl ether with additive exhibited complete wetting in dry nitrogen from 150° to 250° C (302° to 482° F). Table III summarizes the dip cell results. Reproducibility in this experiment was about $\pm 5^{\circ}$ C (9° F).

TABLE III. - SUMMARY OF DIP CELL

EXPERIMENTS IN DRY NITROGEN

Temperature range, $^{\circ}$C ($^{\circ}$F)		
Poor wetting	Partial wetting	Complete wetting
150 to 195 (302 to 383)	195 to 200 (383 to 392)	200 to 250 (392 to 482)

The poor wetting region of the base fluid does correspond roughly to the high wear region of the base fluid in dry nitrogen. It was obvious that an oleophobic or autophobic film was present on the test specimens. At least, a film was produced on the test specimen after contact with the fluid which caused the poor wetting condition of the polyphenyl ether. The nature and source of the film is not known.

It is possible that an oleophobic film was formed on the test specimens during the cleaning procedure. However, all specimens were thoroughly cleaned until they could be completely wetted with distilled water. This rules out the possibility of an initial contaminating organic film. A water film could remain even though specimens were dried in a desiccator. Fein (ref. 12) found that the humidity of the ambient atmosphere when the specimens were cleaned could have a long range effect on wear. However, it seems unlikely that a water film could remain on the metal surfaces at temperatures to 200° C (392° F).

Secondly, a polar-nonpolar impurity could be present in the polyphenyl ether which would immediately adsorb on the clean metal to form an oleophobic monolayer (ref. 13). This film would eventually desorb as the temperature is increased and then allow wetting by the bulk fluid. A sample of the polyphenyl ether was percolated through activated alumina which should remove any polar impurities. This sample exhibited exactly the same poor wetting behavior as the nonpercolated fluid.

Finally, cyclic ethers have been found to be autophobic on clean steel surfaces (ref. 14). The critical surface tension of wetting γ_c for cyclic ethers was found to be approximately 30 dynes per centimeter. The polyphenyl ether used in this study has a surface tension of 50 dynes per centimeter at 20° C (68° F). Therefore, this fluid will not spread on its own adsorbed monolayer until its surface tension becomes less than γ_c. The surface tension of organic fluids decreases linearly with increasing temperature. The rate of change of surface tension with temperature $-d\gamma_{LV}/dT$ is not known for the five-ring polyphenyl ether. Petke (ref. 14) determined $-d\gamma_{LV}/dT$ for several fluids including two aromatic compounds, 1-bromonaphthalene and bromobenzene. The value of $-d\gamma_{LV}/dT$ for these compounds was 0.0938 and 0.0930 dyne per centimeter per $^{\circ}$C (9.38×10^{-7} and 9.30×10^{-7} N/cm/$^{\circ}$C), respectively. If one assumes a similar value for the polyphenyl ether, its surface tension would approach 30 dynes per centimeter (3×10^{-4} N/cm) at about 210° C (410° F).

The autophobic explanation appears to be the most plausible reason for the poor wetting behavior of the polyphenyl ether in dry nitrogen and perhaps its poor boundary lubrication characteristics below 200° C (392° F).

The wettability additive, because it is highly surface active, would be effective no matter which situation exists. It would readily displace an adsorbed oleophobic or autophobic film, thus changing the critical surface tension of wetting γ_c. It may also lower the surface tension γ_{LV} of the bulk fluid. More work is obviously needed in this area.

Effect of Wettability Additive on the Coefficient of Friction

The coefficient of friction for the polyphenyl ether with and without additive in dry and wet nitrogen, and dry and wet air appears in figures 3(a), 3(b), 3(c), and 3(d), respectively.

A friction coefficient of less than 0.25 was observed for the polyphenyl ether with and without additive in all four atmospheres from 150° to 350° C (302° to 662° F). From 150° to 200° C (302° to 392° F), the additive generally decreased the friction coefficient from 25 to 50 percent. Little effect was observed above 200° C (392° F).

SUMMARY OF RESULTS

A pin-on-disk sliding friction apparatus was used to determine the effect of humidity and a wettability additive (of the halogenated organic acid type) on boundary lubrication of steel with a five-ring polyphenyl ether. The test atmospheres were (1) wet air (RH 50%), (2) wet nitrogen (RH 50%), (3) dry air (<100 ppm H_2O), and (4) dry nitrogen (<20 ppm H_2O). Other conditions included a 1-kilogram load, 17-meter-per-minute surface velocity, and a disk temperature range of 150° to 350° C (302° to 662° F). Test specimens were made of CVM M-50 steel. The major results were as follows:

1. Poor wettability was observed for the polyphenyl ether in dry nitrogen from 150° to 200° C (302° to 392° F) and high wear also occurred in this temperature range.
2. The wettability additive improved the wetting characteristics of the polyphenyl ether in dry nitrogen. The additive decreased wear of the polyphenyl ether in dry nitrogen, increased wear in wet air, and had no or little effect on wear in wet nitrogen and dry air.
3. A relative humidity of 50 percent decreased wear in nitrogen and had little effect on wear in air.

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